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14. ABSTRACT
A team from academia, Air Force laboratories and industry has been assembled to develop a design code for one of the prevailing failure modes in thermal barrier systems used for aero-turbines. The failure mechanism to be addressed occurs in systems with two-phase bond coats and is manifest as abrupt delamination along the interface between the thermally grown oxide (TGO) and the intermetallic bond coat. The code will integrate several important time/cycle dependent phenomena, each with associated constituent models for: interface adhesion, bond coat deformation, sintering in the thermal barrier layer, etc. In this the second year of the project, efforts have focused on experimental characterization of the various layers and the development of hierarchical models, both of which are needed to characterize and define the salient governing phenomena. A previously developed interfacial delamination model is being adapted for this problem, and integration of these efforts will provide the pathway to the TBC design code.

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MEANS II: KNOWLEDGE ORIENTED MATERIALS ENGINEERING OF LAYERED THERMAL BARRIER SYSTEMS (NOMELT)

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Abstract

A team from academia, Air Force laboratories and industry has been assembled to develop a design code for one of the prevailing failure modes in thermal barrier systems used for aero-turbines. The failure mechanism to be addressed occurs in systems with two-phase bond coats and is manifest as abrupt delamination along the interface between the thermally grown oxide (TGO) and the intermetallic bond coat. The code will integrate several important time/cycle dependent phenomena, each with associated constituent models for: interface adhesion, bond coat deformation, sintering in the thermal barrier layer, etc. In this the second year of the project, efforts have focused on experimental characterization of the various layers and the development of hierarchical models, both of which are needed to characterize and define the salient governing phenomena. A previously developed interfacial delamination model is being adapted for this problem, and integration of these efforts will provide the pathway to the TBC design code.

Research Objectives

The main objective of NOMELT is to develop a code that facilitates the more aggressive design of multilayered thermal barrier systems. This code will incorporate the most important underlying micro-mechanical processes and permit industrial engineers to identify key material parameters and to probe the design space for improved systems. A hierarchical set of models is being developed to support the design code, which will predict the performance of a TBC subject to a single, dominant failure mechanism, as defined by the constituent models. The focus of this project is on a code for thermal barrier systems with two-phase bond coats that fail by delamination along the interface between the TGO and the bond coat. The constituent models developed for this purpose will address four specific time/cycle dependent phenomena: (i) evolution of the bond

coat, (ii) variations in the TGO, (iii) changes in the structure and properties of the ceramic top coat, and (iv) interfacial delamination.

The Integrated Materials Design Team

A team of academic, industrial and Air Force scientists and engineers has been assembled to undertake this project. The participants have extensive experience modeling materials behavior and conducting experiments at the variety of length-scales required to accurately describe multi-layered thermal barrier systems. The PIs (Hemker, Evans, Hutchinson, Pollock, Smith, Srolovitz) have a record of collaborating on projects involving multi-scale phenomena and have all contributed to the advancement of thermal barrier systems through mutual exchanges formulated at various international workshops on this topic. Our primary industrial contact has been Mike Maloney at Pratt & Whitney. This MEANS II program co-organized an international TBC workshop at UCSB in January of 2006 with participation from the MEANS PI's, postdocs and students as well as industrial partners (primarily P&W and GE) and Air Force personnel. Two additional PI-only meetings were held in October 2005 and October 2006 and used to ensure connectivity within the team.

Results from the second year of this study

Wei and Hutchinson had previously developed a model for the steady-state extension of cracks along metal/oxide interfaces [1]. That model uses an embedded process zone (EPZ) to represent the bond rupture characteristics at the crack front, allows plastic deformation to occur on either side of the interface, and has subsequently been modified to permit inclusion of a plasticity length scale. Hutchinson, through continued collaborations with Wei and Evans, is in the process of simulating delamination along the TGO/bond coat interface using a modified version of the Wei Hutchinson model. The EPZ parameters for this code are based on Smith's first-principles results for W_{sep} and $\hat{\sigma}$, and the plasticity length scale is being measured through nanoindentation experiments on the near-TGO γ-Ni layer by Hemker. Additional modifications to this code will depend on the results of evolution studies that are currently underway.

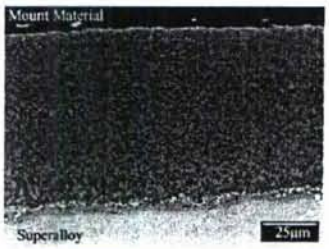
Bond coat evolution. The properties needed to incorporate bond-coat-governed phenomena into the design code include: (a) phase compatibility and interdiffusion, (b) phase transformations, (c) thermo-mechanical properties and (d) gettering of impurities, especially sulfur. Pollock and co-workers at the University of Michigan have conducted thermal cycling experiments and performed SEM and microprobe studies of as-received and thermal cycled bond coats. Hemker and co-workers at Johns Hopkins are complementing the microprobe study with conventional, analytical and high resolution TEM and micro-scale mechanical properties experiments.

We are focusing on NiCoCrAlY bond coats, which additionally contain Y, Si and Hf. Cylindrical laboratory samples of this bond coat on PWA 1484 were prepared at Pratt & Whitney. Studies on bond coat evolution have examined the volume fractions and lattice parameters of the B2 and γ phases in the as-deposited condition and after annealing at 890°C, 950°C, 1000°C, 1050°C, 1100°C and 1150°C. Detailed microprobe studies have

focused on the γ layer that forms just below the TGO, as delamination occurs along this interface. Additionally, characterization of the bond coat, in small pieces of airfoil from blades removed from service at the end of life, has been conducted.

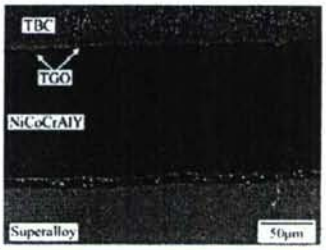
Figure 1 compares the structure of the bond coat in the as-deposited condition with two blade sections which experienced lower temperatures/higher cycles and higher cycles/ lower temperatures. With time during service it is apparent that Al is depleted from the bond coat, due to the formation of the Al_2O_3 TGO. As a result, the volume fraction of the B2 NiAl-based phase decreases, particularly within the upper 20 microns of the bond coat, and the Al content of the γ layer just beneath the TGO also decreases. The volume fraction of the B2 phase was measured to decrease as the exposure temperature increased.

As-deposited (Bar J)



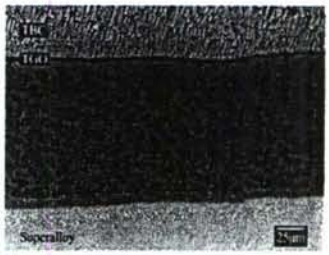
	V_f γ (%)	V_f B2 (%)
Top 10μm	45.5	54.5
Top 20μm	42.3	57.7
Total Thickness	49.2	50.8

Low T / High Cycle Blade



	V_f γ (%)	V_f B2 (%)
Top 10μm	86.8	13.2
Top 20μm	78.5	21.5
Total Thickness	64.0	36.0

High T / Low Cycle Blade



	V_f γ (%)	V_f B2 (%)
Top 10μm	70.0	30.0
Top 20μm	48.7	51.3
Total Thickness	47.6	52.4

Fig 1: Comparison of initial coating structure and evolution of phases with cycling in two turbine airfoil samples.

TEM was used to characterize the microstructure of NiCoCrAlY bond coats at different stages of thermal cycling as well as bond coats that had seen service in commercial engine environments. Traditionally, NiCoCrAlY coatings are considered to consist of a NiAl based β-phase and Ni solid solution γ-phase. The latter however, contained fine, coherent precipitates of γ′ (Ni_3Al) ~7 nm in size in the as-prepared bond coat and ~24 nm for the thermal cycled specimen (Figs. 2(a) and (b)). The unreasonably slow rates of coarsening suggests that γ′ is not a stable, equilibrium phase at the peak thermal cycling temperature of 1100°C but rather forms during cooling. Phase equilibria calculations on the Ni-Co-Cr-Al quaternary provide some evidence for this but differential thermal analysis (DTA) experiments are underway to determine the precise γ′ solutionization temperature.

In single-phase (Ni,Pt)Al bond coats superalloy-bond coat interdiffusion can lead to a martensitic transformation that has a critical effect on the failure mechanism of the TBC system. Fig. 2(d) shows that with thermal cycling the β-phase in the as-prepared NiCoCrAlY bond coat (Fig. 2(c)) also transforms to $L1_0$ martensite. TEM *in-situ* heating

of the martensite revealed complete transformation to the β-phase at ~100°-140°C while the reverse transformation took place at ~50°-80°C on cooling. Therefore, although the strain produced by the volumetric change of the transformation is expected to be large the low transformation temperature together with the fact that NiCoCrAlY coatings are 'two phase' materials strongly indicate that martensite is not significant in this system.

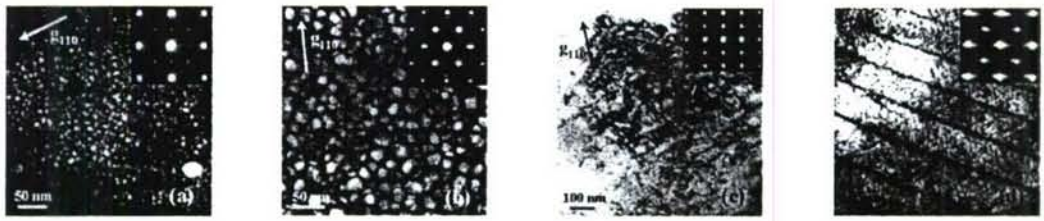


Fig. 2: Dark field images of γ' precipitates in the γ-phase of (a) the as-prepared bond coat and (b) thermal cycled bond coat. β-phase grains in the as-prepared bond coat (c) transform to martensite after thermal cycling (d).

TBC systems based on NiCoCrAlY bond coats typically fail by delamination at the thermally grown oxide (TGO)-bond coat interface. Sulfur impurities greatly reduce adherence and lead to premature oxide spallation. Reactive elements such as Y and Hf in the coating are thought to improve lifetime by forming stable sulfides and / or oxy-sulfides but direct evidence for this has been hard to come by. Using Scanning TEM (STEM) we have detected S precipitates within 'pegs' in a NiCoCrAlY bond coat that had seen service in a commercial engine (Fig. 3). The 'peg' (i.e. regions of abnormal oxide growth) in this case appears to have formed by entrapment of a Y-rich intermetallic by 'folding' of the surface during shot peening. The result highlights the role of reactive elements in gettering the S before it reaches the TGO-bond coat interface and causes embrittlement.

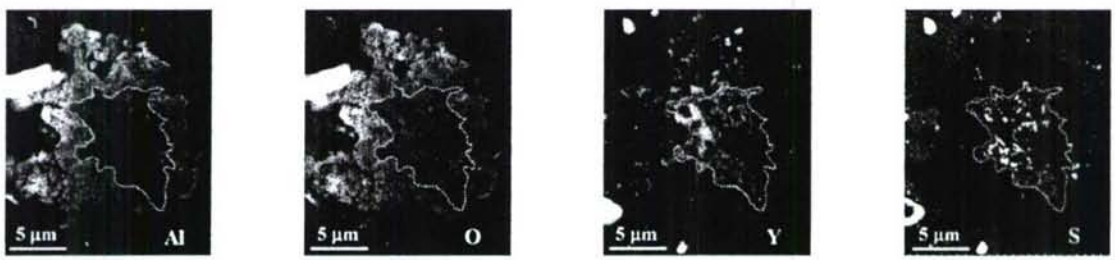


Fig. 3: STEM maps of (a) Al, (b) O, (c) Y and (d) S distribution within an oxide peg.

The fracture toughness of an interface between a brittle ceramic and a plastically deforming metal is heavily determined by the plastic deformation of the metal at the crack tip. Strain gradient theory allows one to calculate the behavior of the material based on experimental measurements of the so called length scale parameters. These material parameters can be accessed by nanoindentation experiments. Microstructural investigations have shown that the TGO formation depletes the region in the top coat close to the interface from Al, leading to the formation of a γ-Ni layer.

Cross sections of thermally cycled and isothermally annealed burner rig bars as well as sections from a turbine blade (in service) were mechanically polished to a mirror finish. The samples were then carefully electro- polished to reduce the roughness and remove any damage caused by the mechanical polishing. Nanoindentation experiments have been carried out by Eberl at UCSB with a Hysitron Tribodenter equipped with a scanning module that allowed him to visualize the surface before indentation. The indentations were conducted with a Berkovich and a cubed corner tip. The γ-phase in NiCoCrAlY exhibited considerable pile-up during indentation. Furthermore the load-displacement curves (Fig. 4) show an atypical decrease from the high slope at the beginning of the indentation. The interpretation of these results, as well as further experiments with the group of WD Nix at Stanford University, are underway.

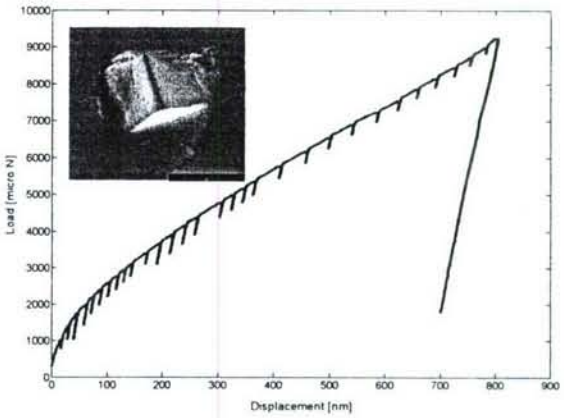


Fig. 4: Nanoindentation: load displacement curve. Inlay shows SEM micrograph of an indented area with an area of 1.2 micron.

TGO evolution. In collaboration with professor Kagawa at Tokyo University, a scanning fluorescence spectroscopy method has been used to characterize the ambient temperature stress state in the thermally-grown oxide. This method allows large area measurements. The objective has been to ascertain regions of the TGO that are most susceptible to through-thickness cracking. Materials provided by Pratt & Whitney and Kawasaki Heavy Industries have been investigated. In both cases, regions of reduced compression have been identified as potential cracking sites, which increase in extent as the system experiences thermal cycling. These regions have been cross-sectioned in the FIB and examined by scanning electron microscopy. The initial finding is the sites exhibiting reduced compression contain various ternary oxide domains in the alumina/YAG phase-field, as well as smaller domains of hafnia. In specimens that have experienced the largest number of cycles, separations are evident between these domains and the surrounding alumina. Such measurements are continuing and should lead to a resolution concerning the source of the TGO cracks that initiate delamination failure of the system.

Top coat evolution. The microstructure and phase stability of TBCs is an important consideration in the study of the reliability of such coatings. In a recent experimental study, Lughi et al., examined the microstructures that result from the sintering of yttria stabilized zirconia (YSZ) TBCs under isothermal conditions and found that adjacent YSZ columns in the coating sinter through coalescence of the feathery features along their length [2]. More importantly, they found that clusters several YSZ columns in size and 'mud-cracking' like patterns were formed after long isothermal anneals of TBCs deposited on bond-coated superalloys. High magnification micrographs of the 'mud-cracks' revealed that these were qualitatively similar to other inter-cluster gaps, and differed only in the size of the gap. These findings pose several interesting questions such as: (1) How does sintering of adjacent YSZ columns lead to the formation of relatively large sized clusters, (2) How do these clusters arrange themselves to form mud-crack like patterns, (3) How do the high temperature

gradients that exist in the coatings in real turbine environments affect sintering/mud-cracking, and, (4) How do the formation of these clusters affect the thermo-mechanical failure of TBC systems. To address questions (1) and (2) Srolovitz is modeling the evolution of the microstructure of the YSZ coating due to the sintering of individual columns via an approach that incorporates the energetics and kinetics of sintering of individual pairs into a framework for evolution of large numbers of columns.

The YSZ columns are idealized as cylinders with a radially symmetric saw-tooth profile used to describe the 'feathery' protrusions of the YSZ columns (Fig. 5). Following the experimental results described in [2], only the saw-tooth protrusions of adjacent columns are allowed to sinter with one another. The chosen density of YSZ columns is distributed at random, and a Monte Carlo simulation is performed to ensure that the cores of the YSZ columns with radii R_c (see Fig. 5) do not overlap. Note, however, that there is initial overlap between the saw-tooth like protrusions of some of the adjacent column pairs. This procedure is used to obtain the initial distribution of columns. The sintering of adjacent columns is described by a thermodynamically motivated variational principle, wherein the rate of change of the free energy of the system (surface energy of column facets + grain boundary energy + elastic strain energy) is equated with the rate of dissipation (from surface and grain boundary diffusion). The shape idealization employed enables a relatively simple, analytical description of the sintering process. The YSZ columns are assumed to be rigidly attached to the substrate and the energy required to bend the columns so that their upper portions can sinter is assumed to be the only contribution to the strain energy. Adjacent columns are allowed to sinter via rate laws obtained from this approach. The shrinkage rates of all possible pairs containing a given column are vectorially added to obtain a displacement rate for that column. Note that these displacements refer to the deflections at the top of the columns. These displacement rates are numerically integrated to obtain column deflections at any given instant of time. A sample size of 2500 columns with periodic boundary conditions is employed.

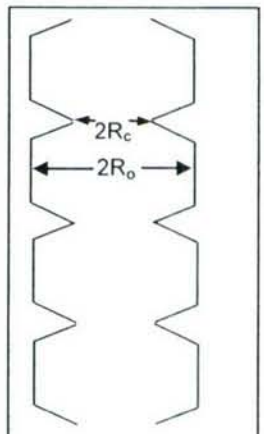


Fig. 5: A schematic representation of the shape assumed for the YSZ columns

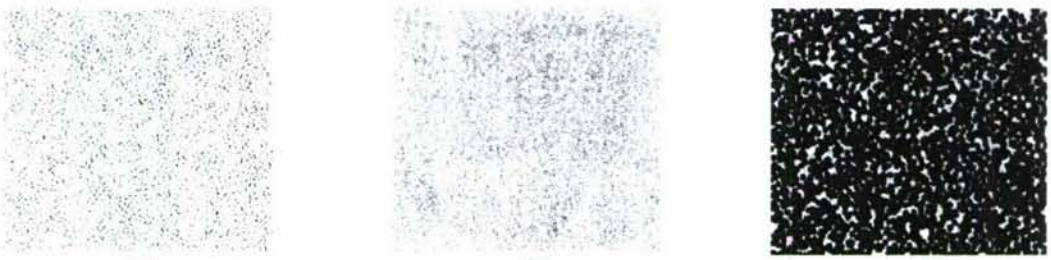
The kinetics of cluster formation depends upon the allowed extent of overlap. To explore the kinetics of cluster formation in different regimes, we chose two different initial configurations. The first configuration is generated using the initialization procedure described above. To generate the second configuration, the core radii of the columns (R_c) are arbitrarily reduced to a lower value after the initial distribution is realized using the Monte Carlo procedure. In addition, a lower value is assumed for the ratio R_O/R_c as compared to the first configuration. These two steps have the effect of decreasing the extent of initial overlap and increasing the surface diffusion lengths, thus allowing for slower densification / sintering rates as compared to the first configuration. These two configurations allow us to explore two different clustering regimes, an early-time regime where sintering is restricted essentially to initially overlapping pairs and only small clusters are formed (examined using the 2nd configuration), and a late-time regime where large-sized clusters with significant inter-cluster gaps are formed (examined using the 1st configuration).

(a) (b) (c)

Fig. 6: Plan view of the microstructures generated after exposure to the sintering temperature for normalized times (a) t= 0, (b) t = 750 and (c) t=1500. The density of the non-overlapping cores is 0.4. The initial microstructure in (a) is superposed on the microstructure in (c) to allow easy comparison of the two.

Coating microstructures that were generated using the 2nd configuration as initial condition and after varying times of exposure to the sintering temperature are shown in Fig. 6. The initial distribution (Fig. 6a) corresponds to strings of loosely overlapping columns with no well-defined cluster size. Upon sintering, small tightly bound clusters of neighboring columns, separated by larger gaps are generated. The gaps, however, are small and well-distributed through the microstructure. Note that the white space shown in these figures correspond to the two-dimensional porosity in the plane of view and not the overall porosity. The overall solid volume and porosity are, of course, conserved during the sintering process.

Coating microstructures generated using the 1st configuration as the initial condition and after varying time periods of sintering are shown in Fig. 7. Again, sintering produces tightly bound clusters and inter-cluster gaps. In contrast to the previous case, however, we see much larger clusters (containing 30-40 columns or more) that are separated by large gaps. Figure 7c clearly demonstrates how gaps sizes and cluster sizes are increased with prolonged sintering. The cluster sizes and gap sizes seen in Figs. 7 (b,c) are similar to those seen in the experiments. Figs. 6 and 7 clearly indicate that two different clustering regimes, one where tightly bound small clusters essentially comprising neighboring columns are formed, and the other where large clusters and inter-cluster gaps are observed characterize the clustering process. It appears that there is a distinct time scale associated with this large scale clustering.



(a) (b) (c)

Fig. 7: Plan view of the microstructures generated using the 1st configuration as the initial condition, after exposure to the sintering temperature for normalized times (a) t= 0, (b) t = 750 and (c) t=1500. The density of the non-overlapping cores is 0.4. The microstructure in (b) is superposed on the microstructure in (c) to help visualize the evolution of cluster and gap sizes with sintering.

These results demonstrate that sintering of columns can result in a microstructure that is characterized by cluster sizes of tens of columns and large inter-cluster gaps. The inter-cluster gap size and cluster size grow with prolonged sintering, and eventually attain an asymptotic value as an equilibrium distribution is attained. Two different clustering regimes, and associated time scales, related to the formation of small clusters and inter-neighbor sintering and the formation of large-sized clusters and inter-cluster gaps can be identified. The formation of large clusters will result in areas of high elastic modulus, resulting in a loss of through-thickness strain compliance in the coating. The large thermal gradients that are present in actual coating conditions, coupled with the gradients in elastic modulus that now exists in the coating can lead to the formation of cracks in the coating.

One of the most important material properties needed for the life time prediction model for TBCs are the mechanical properties of the YSZ top coat. Since a direct measure is not possible due to the brittle behavior of the porous ceramic, bending tests with thin bond coat beams covered with YSZ top coat were carried out under fixed beam conditions.

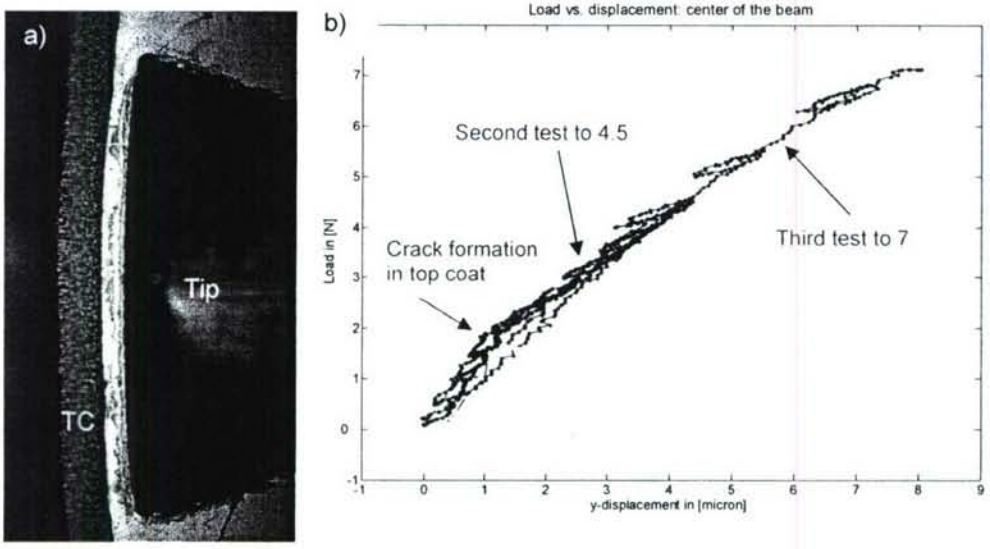


Fig. 8: a) Optical micrograph shows the free standing bond coat (45 micron thickness) with the YSZ top coat (TC, 100 micron thickness) and the cut out region beyond with the actuated tip. b) Load-displacement curve of the bending test shows the change in structural stiffness after crack formation in the YSZ top coat.

A 500 micron thick slice of a YSZ coated burner rig bar in the as processed condition was micro-EDM machined to make the freestanding beams shown in Fig. 8. The TBCs layers are influenced by the presence of the bond coat so 30-50 microns of bond coat were left under the top coat. Bending experiments were carried out by pulling at the beam centers with a special hook-like tip mounted to a microsample testing setup. The strain in the top coat was measured by the use of an optical microscope in combination with a digital camera and a custom full-field digital image correlation software package. The beams were loaded and unloaded until crack formation and finally catastrophic spallation of the TBC occurred. The post processing of the images made it possible to track the strain at each point in the top coat as well as the position of the neutral axis in the bilayered beam.

This data is being used in combination with Finite Element calculations (conducted by Evans at UCSB) to determine the non-linear elastic properties and the toughness of the YSZ coating. Due to the achieved high local strain resolution, further experiments will be carried out with the aim to measure the fracture toughness of the interface between the top coat and the bond coat. These results could be used as comparison to theoretical results from numerical simulations described in the next section.

Interfacial properties. The adhesion of the TGO/bond coat interface has a central role in the design model. To understand trends and to establish the effects of alloy composition and segregation, efforts are underway to: (a) calculate using first principles the work-of-separation of the interface (Smith) and (b) experimentally measure the evolution of the toughness of TGO/bond coat interfaces (Evans, Hutchinson and Hemker).

The need to know the Al activity in γ-Ni is fundamental to our research on adhesion of the thermally-grown oxide Al_2O_3 to γ-Ni. Smith has determined, for the first time, activities of dilute Al solid solutions in Ni with a first-principles approach. Both thermal lattice vibration and electronic contributions to free energies were considered and compared. Vibrational contributions were found to dominate the temperature dependencies of the free energies, although electron thermal effects were also found to be significant. These calculations show opposing temperature trends for the formation enthalpies and entropies, leading to a partial cancellation of their role in the overall energetics. Nevertheless, their remaining temperature effects are strong. Over the temperature range, $400\ K < T < 1700\ K$, the Al activity coefficient varies by 15 orders of magnitude, due to the relative strength of Al-Ni and Al-Al bonds. By comparison, the Ni activity coefficient varies by less than 4% over the same range. The results of this study compare well with available experimental data, and the thermodynamic principles elucidated from the calculations have allowed us to provide a fundamental interpretation of these results.

First-principles calculations conducted over a broad range of atomic configurations have also been used to determine the phase diagram and work of separation for Ni/Al_2O_3 interfaces. Seven interfacial phases have been identified. The results reveal that the strongest (O-rich) phases derive their strength from ionic Ni-O bonds across the interface, reminiscent of NiO. The Al-rich phases are also strong, exhibiting a mix of Ni_3Al-like and Al_2O_3-like interfacial bonds. The stoichiometric interfaces are the weakest since they are formed from the ground-state $Al_2O_3(0001)$ surface.

Wedge indentation experiments by Evans and co-workers indicate that the energy density in the Pratt & Whitney thermal barrier system becomes supercritical beyond a critical TGO thickness. The wedge indentation experiments resulted in large scale spallation with edge sites where the TGO and top coat separated from the bond coat and substrate but did not spall. Hutchinson and Evans have derived buckling models that incorporate measured local geometry and the elastic properties of the various layers in a way that allows them to determine the fracture toughness of the TGO – bond coat interface. Preliminary results suggest a value of $\Gamma = 60\text{-}80 Jm^{-2}$.

Design of a new thermal cycling test. Following cyclic oxidation, many TBC coatings fail by catastrophic spalling at the bond/coating TGO interfaee. These failures often occur in a delayed mode after cooling to room temperature, indicating the moisture may play a role in the final failure process. However, because of the catastrophic character of the final failure, it is difficult to study the early stages of crack initiation and propagation. While there must be flaws that enable a state of tension to develop in the TGO, the features responsible for initiation of the failure are still not well understood due to the character of the final failure. For this reason, UM has developed a new "hot spot" approach to cyclic oxidation. It is worth noting that catatrophic spalling of the TBC in blades often occurs near "hot spots" that develop due to the aero-thermal design of the blade and cooling passages.

The hot-spot apparatus is shown in Fig. 9. Induction coils located at the central region of the cylindrical bar provide a high degree of local heating that decays with distance from the coil, as shown in Fig. 9. Note that a sample tested in the apparatus has experience TBC spalling over the region that experienced a temperature in excess of 1050°C. Typical one hour cycles with relative slow heating and cooling have been conducted. This hot spot test is an efficient means of assessing coating behavior over a spectrum of temperatures and will be utilized in the upcoming year for more detailed studies of failure initiation.

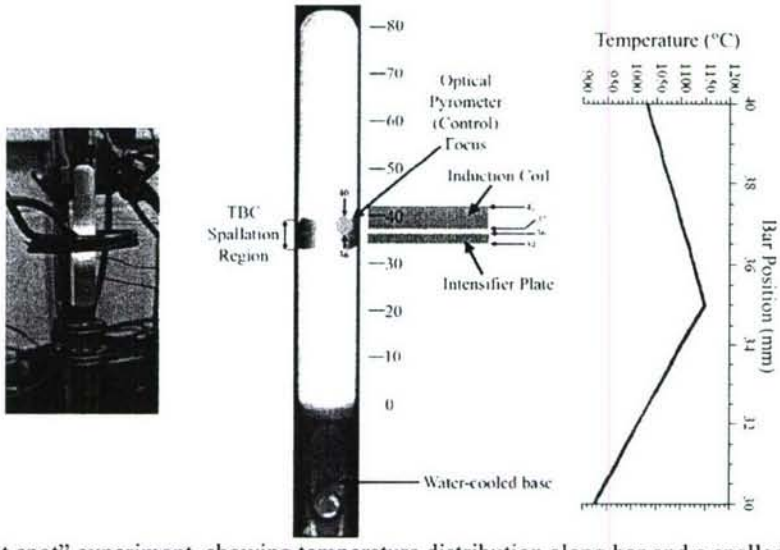


Fig. 9: The "hot spot" experiment, showing temperature distribution along bar and a spalled ring of TBC at the hot spot, above 1050°C

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Transitions

- Dr. Brian Tryon, who worked on this project as a postdoc at the University of Michigan, has been hired by the Coatings group at Pratt & Whitney.

Awards Received

- Professor Evans received the 2006 Henry Marion Howe Medal (ASM International)
- Professor Evans elected as Fellow, Royal Academy of Engineering